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Technologies for the Recovery of Solvents from Hazardous Wastes

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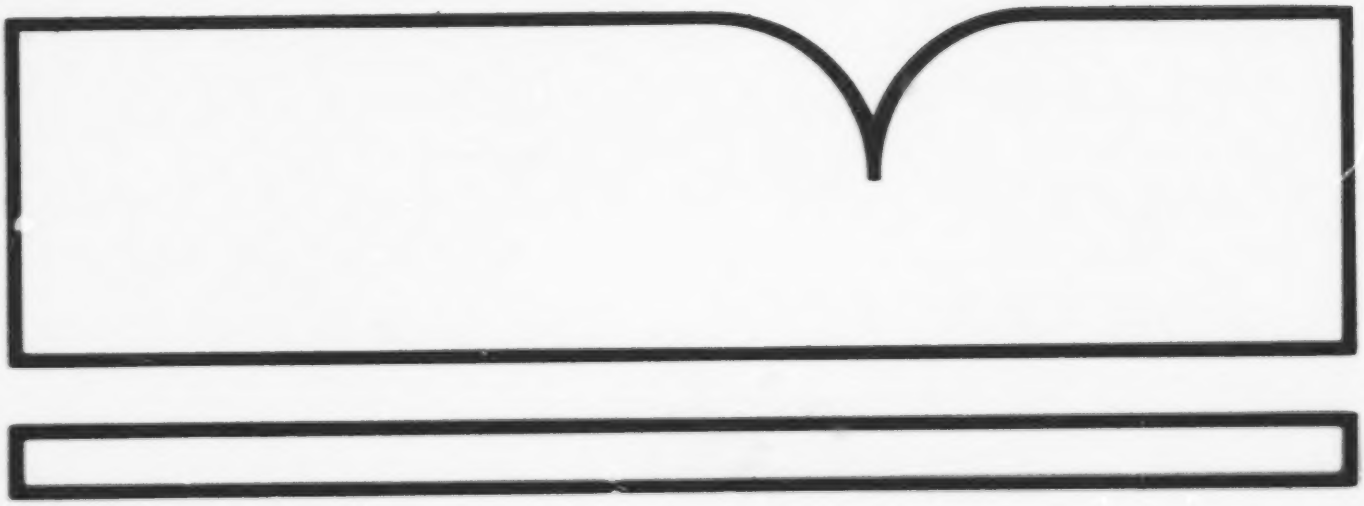

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Technologies for the Recovery of Solvents from Hazardous Wastes

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ABSTRACT

About 1.5 billion gallons of hazardous waste solvents were disposed of to the land in 1985. The Hazardous and Solid Waste Amendments (HSWA) of 1984 required that EPA prohibit the direct land disposal of these materials after November 8, 1986. Alternatives to direct land disposal include source reduction, destruction, treatment, and recovery. This paper describes several technologies that can be used to recover solvent materials from hazardous wastes. Such technologies include evaporation, distillation, solvent extraction, critical fluids processing, steam and air stripping, and carbon adsorption. Data from USEPA studies are presented on recovery efficiencies for several of the unit processes.

INTRODUCTION

In 1981, EPA estimated that three to five billion gallons of hazardous waste spent solvents are generated annually in the United States (1). Table 1 lists the EPA Resource Conservation and Recovery Act (RCRA) spent solvent code designations and waste sources (2). Besides spent solvents containing these compounds, still bottoms from the recovery of these spent solvents are also included under each of the five waste codes.

Under the terms of the Hazardous and Solid Waste Amendments of 1984 (HSWA), Congress required that EPA promulgate regulations to restrict the direct land disposal of spent RCRA solvents by November 8, 1986. If EPA had not met its statutory requirement, the land disposal of any spent hazardous waste solvent material would be automatically prohibited as of that date. EPA estimates that, in 1985, approximately 1.5 billion gallons of spent RCRA solvents were disposed of to the land. The predominant methods for land disposal of spent solvent materials are, in order of usage, deep well injection, landfills, and surface impoundments.

There are four principal approaches that can be employed as options to the direct land disposal of waste solvents. In descending order of preference, these approaches are:

1. Elimination or reduction of waste generated.
2. Recycle/Reuse, including use as a fuel substitute.
3. Destructive treatment, including thermal oxidation.

TABLE 1

REGULATED WASTE SOLVENTS

EPA WASTE CODE	SOLVENTS
F001	The following spent halogenated solvents used in degreasing: Tetrachloroethylene, trichloroethylene, methylene chloride, 1,1,1-trichloroethane, carbon tetrachloride, and chlorinated fluorocarbons; all spent solvent mixtures/blends used in degreasing containing, before use, a total of ten percent or more (by volume) of one or more of the above halogenated solvents or those solvents listed in F002, F004, and F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.
F002	The following spent halogenated solvents: Tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, chlorobenzene, 1,1,2-trichloro-1,2,2-trifluoroethane, ortho-dichlorobenzene, trichlorofluormethane, and 1,1,2-trichloro-ethane; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above halogenated solvents or those listed in F001, F004, or F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.
F003	The following spent non-halogenated solvents: Xylene, acetone, ethyl acetate, ethyl benezene, ethyl ether, methyl isobutyl ketone, n-butyl alcohol, cyclohexanone, and methanol; all spent solvent mixtures/blends containing, before use, only the above spent non-halogenated solvents; and all spent solvent mixtures/blends containing, before use, one or more of the above non-halogenated solvents, and, a total of ten percent or more (by volume) of one or more of those solvents listed in F001, F002, F004, F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.
F004	The following spent non-halogenated solvents: Cresols and cresylic acid, and nitrobenzene; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above non-halogenated solvents or those solvents listed in F001, F002, and F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.
F005	The following spent non-halogenated solvents: Toluene, methyl ethyl ketone, carbon disulfide, isobutanol, pyridine, benezene, 2-ethoxyethanol, and 2-nitropropane; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above non-halogenated solvents or those solvents listed in F001, F002, or F004; and still bottoms from the recovery of these spent solvents and spent solvent mixtures.

4. Treatment for removal or reduction of toxic or flammable constituents prior to land disposal.

EPA recently estimated that 0.55 billion gallons of spent solvents are recycled, with 80 percent of this recycling occurring at the site of waste generation. Approximately 15 percent of the waste that is recycled off-site is used as fuel (3).

The relatively small amount of solvent recycling that is presently being practiced indicates that there is ample opportunity for expansion of this management technique. In particular, as the land disposal restriction regulations are now in effect, the decreasing availability and increasing cost of alternative waste management approaches will lead to more extensive practice of source reduction and recycling.

This paper is concerned with technologies that can be used to recover solvents from these hazardous waste streams. The focus will be on recovery of solvents for reuse as solvents. However, the technologies that will be described can also be applied to the recovery of solvent based materials for use as fuels.

The format for presentation will be to deal with wastes in order of their concentrations beginning with the highest solvent concentration wastes and moving through to the liquids with the lowest solvent concentrations. Some of the technologies that are described for high concentrations may be applicable for the other materials after some initial treatment to enhance concentration.

HIGH SOLVENT CONCENTRATION WASTES

For purposes of this discussion, high concentration solvent wastes are those with a concentration of from 10 to 100 percent. Techniques that have applicability to these wastes include evaporation, distillation, solvent extraction, and critical fluids extraction.

Evaporation

Evaporation is the removal of a solvent (including water) as a vapor from a solution or a slurry (a pumpable solid-liquid mixture). Agitated thin-film evaporation relies on the exposure of a large surface area of heated waste to atmospheric or vacuum conditions to enhance the separation of more volatile constituents. Waste is spread on the walls of a cylindrical or tapered tube by an assembly of wiper blades. A heated outer shell elevates the temperature of the waste. Figure 1 shows one design of a vertical thin-film evaporator. Waste is fed into the top, moves down the inside walls and exits at the bottom. Evaporated material exits overhead and goes to a condenser. The unit is heated by steam or hot oil circulating through the walls.

Steam pressure in the jackets can reach 200 psig, while heated oil fluid temperatures can be as high as 650°F. Residence time for the waste material can be controlled from 3 to 100 seconds.

There are two major advantages from waste agitation by the wiper blades. First, it creates turbulence in the liquid, increasing the transfer of heat to the waste and of volatiles to the waste surface, where they evaporate. Second, the wiper blades maintain a more uniform film and even flow of the liquid through the unit, which is particularly important when the waste is viscous and/or contains solids.

Agitated thin-film evaporators (TFE) are the "work horse" of many solvent waste recycling firms. They provide the first step in the processing of those solvent wastes which are suitable for recycling, i.e., those with a sufficiently high percentage of recoverable organics (typically over 50 percent). Wastes are treated by agitated thin-film evaporation to separate the solvents from non-volatile solids and high boiling organics (bottoms). The overhead fraction is often processed further by distillation, water extraction, etc., prior to reuse. Currently, some solvent-bearing bottoms are used as a supplemental fuel. These bottoms should also be amenable to incineration.

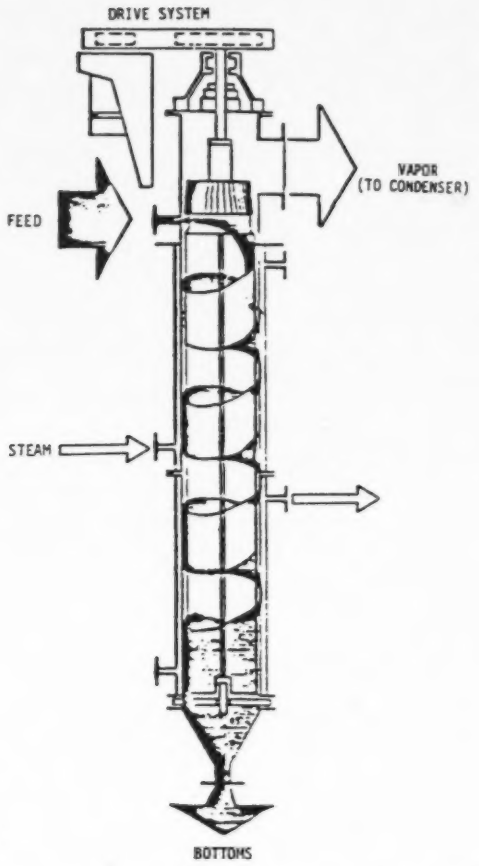

FIGURE 1. FLOW PATH OF AGITATED THIN-FILM EVAPORATOR

Thin-film evaporators can be used to treat liquid wastes and sludges of moderate to low solids content. Waste viscosities generally cannot exceed 100 poises and the size of solids in a sludge is limited by the clearance of the agitator blades (typically less than 2.5 mm). Reactive wastes, such as those which will polymerize, cannot generally be treated using thin-film evaporators because of the elevated temperatures. However, some agitated thin-film evaporators are designed to minimize the waste residence time in order to reduce this problem.

The USEPA recently sampled a thin-film evaporator at a recycler which uses the process for the treatment of paint and lacquer thinners. The thinners contain 2-20 percent dissolved paint resins plus suspended pigments and such nonhalogenated solvents as alcohols, ketones, esters, and aromatic hydrocarbons. Overhead product is sold back to the customers without high boiling components, such as xylene. Bottoms product is maintained in a pumpable state and sold as a waste fuel with less than 30 percent solids and a heating value of up to 16,000 Btu/lb.

The TFE unit has 20 square feet (109 m^2) of heated surface area and a steam heated jacket. The jacket is carbon steel, while the shell is comprised of 304 stainless steel. The blades have variable clearance and are operated at a speed of 300 rpm. The unit is capable of operating under vacuum conditions up to 28 in Hg (9,670 kg/m^2).

A one-day sampling campaign was run on the unit while it was processing a waste paint and lacquer stream with RCRA designation F003/F005. The waste feed

was a greyish-brown homogenous liquid containing primarily toluene (22%), acetone (19%), and methyl ethyl ketone (18%). Other characteristics of the feed stream are shown in Table 2.

The unit processed 1,530 gal (5,792 L) of feed material over a 6.75 hour period (3.8 gpm or 14.3 Lpm) and generated 830 gal (3,142 L) of distillate product and 688 gal (2,604 L) of still bottoms. Other operating conditions were:

Steam pressure: 140-160 psig
Steam temperature: 361-370 °F
Overhead product temperature: 125 °F
Bottom temperature: 138-140 °F
Vacuum: 13-23 in. Hg

Results of products and bottoms analyses for this test are presented in Table 2. The clear, colorless distillate product stream (for which triplicate field samples were collected) was mainly toluene (24 percent), methyl ethyl ketone (22 percent), and acetone (18 percent). The still bottoms stream was mainly xylene (21 percent) and problably other grades of mineral spirits (according to the facility). The heat content of the still bottoms stream is slightly higher than the waste feed and is significant at 14,800 Btu/lb (8,220 kcal/kg).

TABLE 2
RESULTS OF ANALYSES FOR WASTE TREATMENT IN THIN FILM EVAPORATER

ANALYTICAL PARAMETER	WASTE FEED	STILL BOTTOMS	DISTILLATE PRODUCT
Organic (all values in mg/kg)			
Total Xylene	66,000	210,000	84,000
Acetone	190,000	5,200	180,000
Ethyl acetate	11,000	< 12,000	12,000
Ethyl benzene	22,000	48,000	28,000
Methyl isobutyl ketone	14,000	5,600	16,000
n-Butyl alcohol	11,000	< 5,500	13,000
Toluene	220,000	81,000	240,000
Methyl ethyl ketone	180,000	57,000	220,000
Isopropanol	76,000	< 5,500	84,000
Fuel			
Heating Value (Btu/lb)	14,698	14,795	NA
Viscosity (cSt)	2.825	26.156	NA
Density (g/cc)	0.8420	0.9370	NA
Flash Point (deg. C)	20	27	NA
Ash Content (%)	0.14	0.59	NA
Carbon (%)	70.62	72.76	NA
Chlorine (%)	0.24	0.17	NA
Miscellaneous			
Water Content (%)	2.94	8.76	3.81

NA = Not Analyzed

Distillation

Distillation is the separation of the constituents of a liquid mixture by partial vaporization of the mixture. The vapor is condensed and recovered. Nonvolatile residues remain behind.

The purity of the condensed overhead is dependent upon the type of distillation unit used. In a simple distillation unit, the waste is heated and the vapors condensed in a single step; an equilibrium exists between the liquid and the vapor stream leaving the apparatus. If the waste contains low boiling components, each will be present in the condensate. And, if there is not an appreciable difference in volatility between the components desired in the overhead and those to be left in the bottoms, each effluent stream will still be contaminated after the distillation is terminated.

To obtain better separation of the components in the overhead and between the overhead and the bottoms, a fractional distillation unit is employed. A schematic diagram for a continuous fractional distillation column is shown in Figure 2. The waste stream is fed in to the middle of the unit. Above the feed point, in the "rectifying section", are equilibrium mixtures of progressively high concentrations of volatile constituents. Progressively higher concentrations of less volatile components are found below the feed inlet. A portion of the condensed vapor is returned to the top of the column as reflux in order to optimize the purity of the product.

The solids content of a distillation stream is a limiting factor since solids may clog the column or the coils in the reboiler. Therefore, many waste recycling firms use thin film evaporators to separate out solids prior to fractional distillation.

Recently, EPA tested a batch distillation unit that was used to process a liquid feed containing a number of organic compounds. The still was a Pfaudler closed top reactor made of glass lined carbon steel with a capacity of 500 gallons. The distillation unit was equipped with a 6 foot high, 10 inch diameter packed column that provided four product cuts. Heat was provided by jacketed steam.

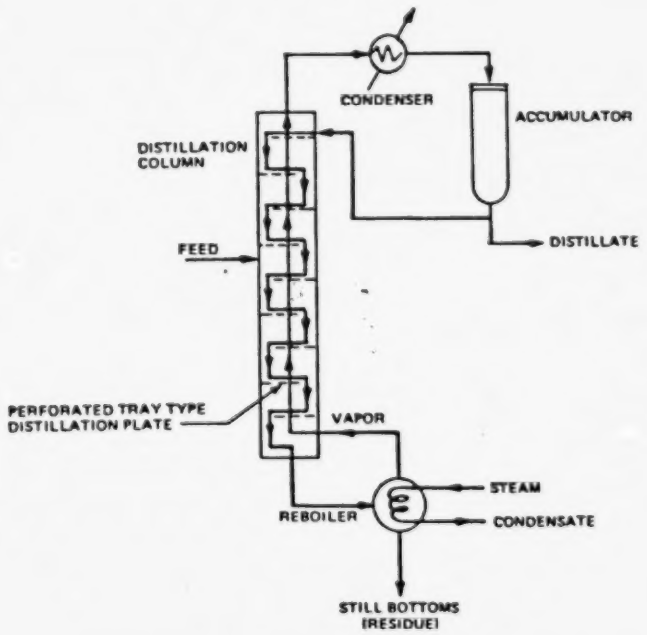

FIGURE 2. CONTINUOUS FRACTIONAL DISTILLATION

The mode of operation was to run the still pot hot and cool the column to the boiling point of the desired solvent product. This procedure provided some refluxing of the solvent vapors back to the still pot.

Table 3 displays data obtained from one batch of liquid material that was processed through the batch distillation unit. The waste was a mixture of the three major components: methanol, methyl ethyl ketone (MEK), and methylene chloride. The water content was approximately four percent. The boiling range of the mixture varied from 44 to 85° C during the distillation process.

TABLE 3
BATCH DISTILLATION DATA (mg/l)

Compound	Boiling Point (°C)	Influent	Still Bottoms	Product Cuts 1	2	3	4
Acetone	56.2	9500	1300	4500	5600	11000	12000
Ethanol	78.3	14000	14000	4200	<500	990	17000
Isopropanol	82.5	37000	98000	8100	8800	37000	72000
Methanol	64.5	300000	230000	160000	190000	180000	190000
Methylene Chloride	40.0	460000	<500	1100000	710000	590000	58000
Methyl Ethyl Ketone	79.6	240000	370000	42000	65000	280000	340000
Toluene	110.6	46000	110000	7300	17000	290000	350000

From Table 3 it can be seen that distillation provides excellent recovery for the low boiling point compounds. The component analysis accounted for 77 percent of the organics. Water was 8 percent of the feed. Some of the chlorinated organics were not identified by the analysis. Further refinement could be achieved through additional product cuts (higher column) or through further distillation of the individual product cuts.

Solvent Extraction

Solvent extraction is a process whereby a dissolved or adsorbed substance is transferred from a liquid or solid phase to a solvent that preferentially dissolves that substance. When the waste to be treated is a liquid (often contaminated water), the process may be called liquid-liquid extraction. The substance transferred is the solute, the treated effluent is referred to as the raffinate and the solute-rich solvent phase is called the extract. For the process to be effective, the extracting solvent must be immiscible in the feed liquid and differ in density so that gravity separation is possible and there is minimal contamination of the raffinate with the extraction solvent.

Solvent extraction can be performed as a batch process or by the contact of the solvent with the feed in staged or continuous contact equipment. The solvent and the wastewater are agitated in one vessel (the mixer) and then transferred to another vessel (the settler) where the phases are separated by density differences. This operation can occur more than once so that there are multiple mixer-settler stages. Another option, which is depicted in Figure 3, is continuous countercurrent contact in a vertical column such as a spray tower, packed tower, or sieve plate tower.

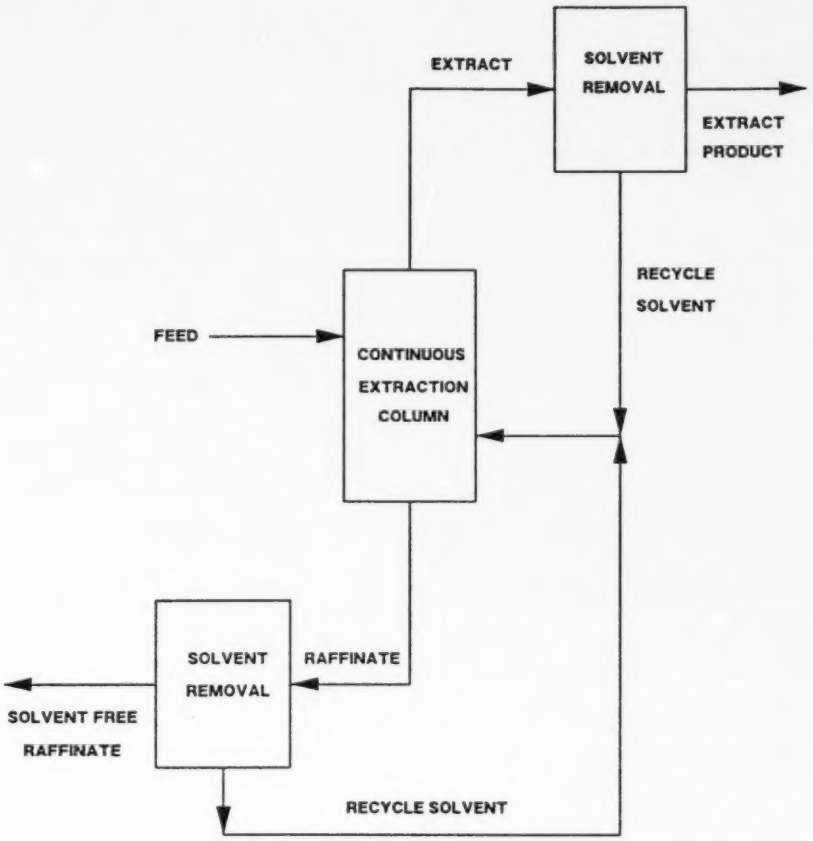

FIGURE 3. CONTINUOUS COUNTERCURRENT SOLVENT EXTRACTION

Solvent extraction is a limited technology in that it is almost always necessary to further treat the raffinate and/or the extract. This technology is typically used when it is not economical or possible to use more standard technologies. The removal efficiency of solvent extraction depends on the solvent, the constituent to be extracted, soil properties (if a constituent is being extracted from soil), the liquid containing the constituent, and the unit operation design. Typical removal efficiencies vary from 80 percent to close to 100 percent.

Solvent extraction has had limited application in hazardous waste processing. Specific examples are removed of phenol from refinery wastewater, removal of dioxins from still bottoms, treatment of wood preserving waste to remove creosote, and soil washing (removal of organic material from contaminated soil).

It is often desirable to reuse the solvent in the solvent extraction process. This is accomplished by using a distillation column to remove the extracted solute molecules from the solvent in a concentrated form and then reusing the distilled solvent.

The cost of solvent extraction is relatively high. The high costs result from initial solvent costs, further treatment of the extract, such as distillation, and disposal costs of the extracted constituents (5).

Critical Fluids Extraction

Critical fluid extraction has potential for use in solvent recovery. Critical fluid technology has been employed for regeneration of activated carbon that has been spent with pesticides and has been used to extract PCBs from oil in laboratory tests.

Critical fluids technology takes advantage of the phenomenon that elevated temperatures and pressures at or near a substance's critical point enhance the substance's ability to act as a solvent. This is due to the fact that the internal energy of the critical fluid is sufficient to overcome the internal bonding of the fluid molecules.

All fluids exhibit critical area properties. Table 4 describes the critical points of a number of common substances.

TABLE 4
CRITICAL POINT DATA

SUBSTANCE	CRITICAL TEMPERATURE (°C)	CRITICAL PRESSURE (atm)
Water	374	218.1
Carbon Dioxide	31	72.9
Oxygen	-83	50.1
Hydrogen	-205	12.8

Given the low energy state of its critical point, carbon dioxide is most often chosen as the working fluid in critical fluid processing. However, the ultimate selection criteria is the miscibility of the solute in the critical fluid.

The extraction process is achieved through the following approach:

1. Compression of the fluid to critical pressure conditions.
2. Heating of the fluid to critical temperature conditions to achieve dissolution of the target material.
3. Controlled decompression of the fluid to allow condensation or precipitation of the target material.

Figure 4 depicts a critical fluid extraction process.

In 1978, under EPA sponsorship, Arthur D. Little applied the supercritical CO_2 solubility concept to the regeneration of granular activated carbon (GAC) used in wastewater treatment. Separate extraction tests were oriented toward the extraction of phenol, acetic acid, and Arachlor from GAC type F-300. Critical fluid temperatures ranged from 55°C to 120°C and pressures varied from 150 atm to 275 atm.

By way of example, Figure 5 describes the desorption of phenol achieved through supercritical processing of the F-300 GAC. Supercritical CO_2 has proven to be an excellent extractant for a number of solvent constituents, including acetone, cyclohexanone, and toluene (6).

Critical fluid extraction has seen limited, but increasing application to hazardous waste cleanup. One reason is that its energy requirements are not as severe as for evaporation and distillation processes. Critical fluid extraction may have some application for treating still bottoms, contaminated soils or spent activated carbon that has been used in solvent processing.

Low Concentration Solvent Wastes

Low concentration solvents are defined here as wastes that have solvent concentrations below 10 percent. Most often these wastes contain liquid solvents

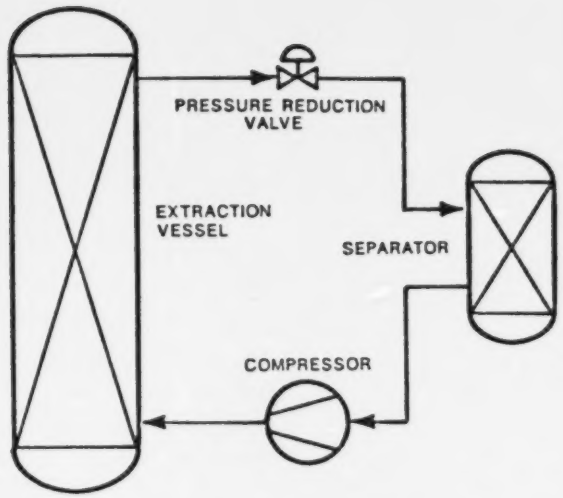

FIGURE 4. SUPERCRITICAL FLUID EXTRACTION PROCESS

in an aqueous medium. In the past, wastes of these low solvent concentrations were not economically recoverable. However, with the new stringent land disposal restriction regulations, management costs for these wastes will increase and recovery of these wastes may be proven to be cost effective. The technologies most applicable for recovery of low concentration solvent wastes are stripping and solvent extraction (discussed above). Other treatment technologies can be used to remove organic compounds from water (e.g., adsorption, biodegradation),

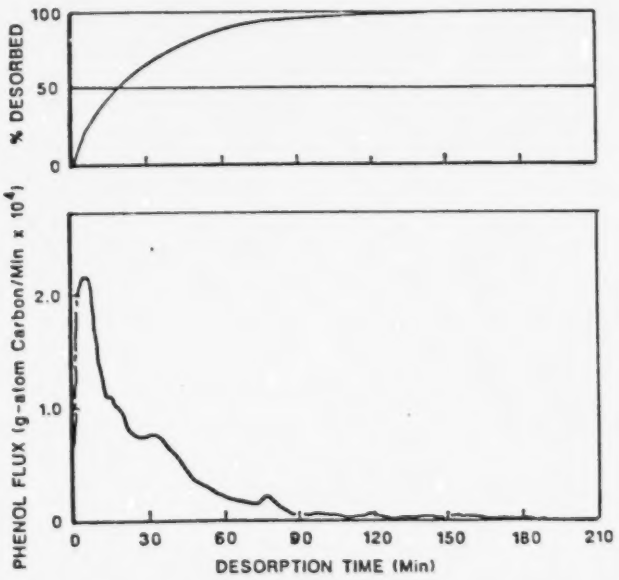

FIGURE 5. DESORPTION OF PHENOL FROM GRANULAR ACTIVATED CARBON WITH SUPERCRITICAL CO_2

b... these either destroy the compounds or require additional, generally expensive steps to recover the organics.

Steam Stripping

Stripping is a physical unit operation in which dissolved compounds are transferred from a liquid into a flowing gas stream. The driving force for mass transfer is provided by the concentration gradient between the liquid and gas phases. In steam stripping, live steam is used as the gas phase. The steam both heats the liquid, which enhances the rate of mass transfer, and carries the released compounds away from the liquid (7).

Steam stripping can be performed in the batch or continuous mode. In the batch process, waste is charged to a boiler and steam is injected directly into the waste. The injection of live steam both heats the waste to volatilize low boiling components and creates turbulence in the waste, thus increasing the rate of volatilization. The gases which are condensed from a steam stripper will contain water along with the more volatile organic components of the waste in the form of a two-phase mixture. This mixture is decanted and the organic component is drawn off for reuse or disposal.

In a continuous steam stripping column, as shown in Figure 6, waste flows down a column while steam flows up. The column is designed to promote heat transfer from the steam to the waste, to cause turbulence in the waste and to create a large waste surface area. All of these properties promote transfer of volatile components from the waste to the gas phase. Different liquid-vapor equilibria exist in the column, with the highest relative concentration of the most volatile components found at the top. As with fractional distillation, the separation of different volatile constituents may be enhanced by refluxing a portion of the condensate.

Steam stripping is generally used to separate insoluble or slightly soluble compounds from water. These compounds are readily stripped because they have a large air to water equilibrium coefficient which increases as the temperature of the waste is raised by the steam. In addition, a high percentage of the mass of the solvent in the condensate will be in the organic phase; only a small portion of the stripped organics are left in the condensed steam.

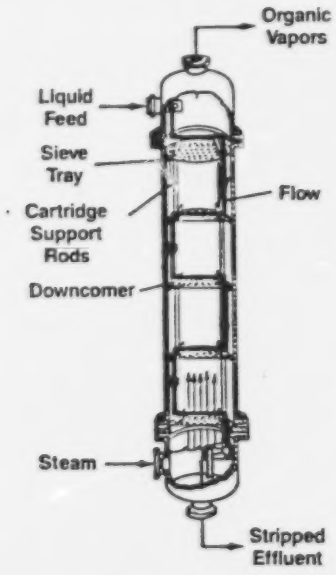

FIGURE 6. STEAM STRIPPING COLUMN - PERFORATED TRAY TYPE

The aqueous phase of a steam stripper's requires disposal, and this requirement must be carefully considered in deciding whether stripping is an appropriate recycling technique. In a few instances, the small amount of water need not be separated from the organic phase and the entire condensate is recycled (8). Generally, however, the aqueous condensate must be treated before discharge to either surface waters or a municipal sewer. While this can be accomplished through a separate process, it usually is done by recycling the aqueous condensate to the influent stream of the stripper. This requires a unit that is oversized to handle the additional load.

One application of steam stripping is the recovery of ethylene dichloride (EDC) from the wastewaters of a chemical manufacturing plant which produces this compound and vinyl chloride monomer (VCM) (8). Wastewaters from production operations and storm water runoff are steam stripped before undergoing further treatment to meet NPDES permit limitations. The feed rate to the stripper ranges from 760 to 950 L/min. Steam at 50 psig is injected into the bottom of this continuous stripping column, while feed enters at the top. The feed-to-steam ratio was typically 20:1. The unit utilizes two condensers: one with cooling water and one with glycol-water at about 2°C.

During two days of testing, the feed stream to the unit was composed of EDC (4,200-7,600 ppm), chloroform (160-440 ppm) and other volatile organics (1-19 ppm). All the condensate, including water, is recycled to the EDC production process. The unit efficiently recycles over 99% of the EDC, leaving a wastewater effluent which contains less than 10 ppm organics (consisting mainly of chloroform).

CONCLUSION

In the final analysis, cost will determine whether a particular spent solvent waste is to be recovered. The HSWA land disposal restrictions limit the management options for waste solvents. As costs for treatment and disposal rise, the economics of solvent recovery will improve. Of course there will always be some solvent wastes that are generated in such low quantity by a facility or contain so many impurities that recovery is not viable. The effect of the HSWA restrictions could be to raise the level of contamination that will be acceptable prior to solvent recovery.

For the time being, evaporation, distillation, and steam stripping will most likely be the technologies of choice for solvent recovery. However, as treatment and disposal costs rise and the technologies are better understood, critical fluids, carbon adsorption, solvent extraction, and, perhaps, other recovery processes will receive increasing use for recovery of waste solvent materials.

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